Coventry University and
The University of Wisconsin Milwaukee Centre for By-products Utilization,
Second International Conference on Sustainable Construction Materials and Technologies
June 28 - June 30, 2010, Università Politecnica delle Marche, Ancona, Italy.
Special Technical Proceedings ed. P Claisse, E Ganjian, F Canpolat and T Naik
ISBN 978-1-4507-1488-4 http://www.claisse.info/Proceedings.htm

Laboratory Ageing Protocols for Asphalt Recycling in Hot Climates

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ABSTRACT

The objective is to produce recycled asphalt pavement (RAP) in the laboratory with controlled properties, typical of those found in hot tropical belts (with residual binders of very low penetration). The RAPs would subsequently be used for laboratory studies on cold recycling. Standard protocols for ageing asphalt mixtures have been developed during the Link BITUTEST and SHRP programmes in the UK and USA, respectively. However, these protocols would require long laboratory ageing times in order to produce the desired properties. Ageing samples at higher temperatures than standard will generate the severely aged RAP in a practical time but risks degrading the binder. This paper presents the results of rheological and physico-chemical measurements of recovered binder from asphalt aged at higher temperatures. Results show that ageing hot mix asphalt at 125°C does not degrade the binder when compared to that aged at 85°C, which is the conventional protocol.

INTRODUCTION

A lot has been done in the developed countries of the world, most of which have temperate climates, to develop cold bituminous emulsion asphalt mixes made from virgin materials. The use of reclaimed asphalt pavements (RAP) under such circumstances has also received attention due to the potential economic, safety and environmental advantages that it offers [PIARC, 2008]. Some of these countries have developed guidelines for the production of cold (recycled) mixes [ARRA, 2001 and Asphalt Academy, 2009]. Results from field trials in temperate climates have been reported to be encouraging [PIARC, 2008]. However, little or nothing has been done to ascertain the performance of such cold mixes and cold recycling of pavements in countries located in the hot climates of the world [PIARC, 2008]. These sustainable methods and materials are needed in road building practices in tropical countries, where available funds for road building and upgrading are increasingly inadequate to meet demand [Nnenna, 2003 and UNECA, 2007].

In developing guidelines for the use of cold recycled asphalt mixes in hot tropical countries (with Nigeria being the case study) the first challenge is sourcing the recyclate. For this investigation, being conducted in the UK, laboratory ageing is required and standard laboratory protocols for long term ageing of asphalt mixtures have been developed during the Link BITUTEST and SHRP programmes in the UK and USA respectively [Bell, 1989 and

Scholz, 1995]. Laboratory ageing under these protocols is conducted at 85°C over 5days, and the assumption is that this is representative of 15 years or more of ageing of pavements in service in temperate regions of the world [Bell 1989 and Scholz, 1995]. Meanwhile, Nnenna [2003] reported that, some roads constructed about 32 years ago in Nigeria have not yet been rehabilitated. Smith and Edward [2001] reported penetration values as low as 6dmm for wearing course and 5dmm for binder course in some asphalt pavements in East Africa. The challenge is to produce the desired RAPs in the laboratory with controlled properties (down to a target penetration of 3dmm), typical of those found in asphalt pavements in hot tropical belts, which would subsequently be used as RAPs for investigation of the suitability of the severely aged RAP materials for cold recycling. Current laboratory ageing protocols use compacted specimens [Airey, 2003] and would require long laboratory ageing times in order to produce the desired properties. Ageing loose samples at higher temperatures than standard will generate more homogeneous [de la Roche et al, 2009] severely aged RAP in a practical time but risks unduly degrading the binder in the mix. Researchers have suggested that this degradation would normally affect the physico-chemical and rheological properties of the binder in the mix [Airey, 2003 and Wu et al, 2007].

In a bid to investigate the extent of degradation of binders in bituminous mixtures when they are aged at relatively high temperatures, this paper studied the physico-chemical and rheological properties of recovered binders, which have undergone relatively high ageing temperatures. The results are compared with those obtained from conventional ageing and the well established conventional bulk binder ageing protocols vis-à-vis rolling thin film oven test (RTFOT) and pressure ageing vessel (PAV) test, for validation purposes. The aim of this investigation is to come up with an ageing protocol that is as quick as possible that would not introduce any significant changes to the properties of the binder in the mix when compared with a standard ageing protocol.

EXPERIMENTAL PROGRAMME

The investigation required verifying and comparing the chemical composition (asphaltene content in line with BS 2000: PART 143:2004), viscoelastic response (rheology in line with BS EN14770:2000) and temperature susceptibility (penetration and softening point in line with BS EN1426:2000 and BS EN1426:2000 respectively) of recovered binders (in line with BS EN12697-4:200) from mixtures immediately after; (1) mixing and compaction (representing short-term ageing), and then subsequently (2) ageing at 85°C, 105°C and 125°C (representing long-term ageing) over 24, 48, 72, 96, 120, 240, 480 and 840hrs in the forced draft oven. Just before being subjected to the long term ageing, the compacted mixtures were loosened in order to ensure homogeneous ageing. The materials were placed in oven trays such that the thickness of the loose materials in the tray was not more than 40mm, also in order to promote homogeneous ageing.

To allow for direct comparison as ageing progresses, the bulk binder was also aged in the Rolling Thin Film Oven (RTFO) and the Pressure Ageing Vessel (PAV) in line with BS EN12607-1:2007 and BS EN14769:2005, respectively. Residues from the RTFO test (subjected to ageing at 163°C for 75minutes) were subsequently subjected to ageing over intervals of 5, 10, 15, 20, 25, 30 and 35hrs in the PAV. The residues were aged in the PAV at 100°C as this simulates what occurs in the tropics [Bahia and Anderson, 1995]. The applied pressure used was 2.1MPa. For rheology, all the binders were tested within the linear viscoelastic range on the Bohlin Gemini Dynamic Shear Rheometer applying the following testing conditions:

- Mode of loading: Strain Controlled
- Temperatures: 10 to 80°C
- Frequencies: 0.1 to 10Hz
- Plate geometries: 8 mm diameter with a 2mm gap (10 to 45°C) and 25 mm diameter with a 1mm gap (25 to 80°C)

MATERIAL TESTED

Granite - This was sourced from Montsorrel Quarry, and chosen because granite is predominantly used for bituminous pavements in Nigeria.

Bitumen - Supplied by Nynas Bitumen. It was a straight run paving grade binder 40/60 of Venezuelan origin.

HMA - Prepared in the laboratory (BS EN12697-35:2004). The mix was compacted to refusal in line with the requirements of ORN 19 and 31 for heavily trafficked roads in the tropics.

TEST RESULTS AND DISCUSSIONS

Results of physico-chemical and rheological tests

Table 1 details the physico-chemical (penetration, softening point, asphaltene and maltene contents respectively) and rheological (complex shear modulus – G* and phase angle – δ) properties of unaged virgin binder, RTFO and PAV aged bulk binders. Table 2 shows the physical properties (penetration, softening point, and penetration index), chemical composition (asphaltene and maltene contents) and rheological (complex shear modulus – G* and phase angle – δ) properties of unaged virgin binder and recovered binders from mixtures at different ageing temperatures.

Table 1. Physico-Chemical and Rheological Properties of Virgin, RTFO and PAV Aged Binders

Condition of Binder	Physical Properties		Chemical Composition		Rheology (@0.4Hz &25°C)	
	Pen (dmm)	SP (°C)	Asphaltene Content (%)	Maltene Content (%)	G* (MPa)	δ (°)
Original	53	51.0	12.5	87.5	0.39	69.9
RTFOT	34	57.0	15.1	84.9	0.99	62.8
PAV 5Hrs	25	61.0	17.1	82.9	1.58	58.2
PAV10Hrs	24	62.4	17.6	82.4	1.93	56.6
PAV 15Hrs	20	64.6	18.7	81.3	2.27	53.9
PAV 20Hrs	21	65.2	17.5	82.5	2.30	53.7
PAV 25Hrs	23	68.0	19.0	81.0	2.95	50.8
PAV 30Hrs	18	67.8	19.9	80.1	3.25	49.6
PAV 35Hrs	17	69.2	23.5	76.5	3.70	48.7

Note: Pen = Penetration, SP = Softening Point, G= Complex Modulus, and δ = Phase Angle*

Table 2. Physical Properties, Chemical Composition and Mechanical Properties of Virgin and Recovered Binders at Different Ageing Temperatures

Time of Ageing	Physical Properties			Chemical Composition		Rheology (@0.4Hz &25°C)	
Unaged and After Mixing							
	Pen (dmm)	SP (°C)	PI	Asphaltene Content (%)	Maltene Content (%)	G* (MPa)	δ (°)
Unaged	53	51	-0.81	12.5	87.5	0.39	69.9
After Mixing	40	59	0.29	15.6	84.4	0.95	61.7
Ageing at 85°C							
24	31	60.0	-0.06	14.9	85.1	0.96	60.5
48	27	64.0	0.39	16.3	83.7	1.59	56.8
120	23	68.0	0.74	19.0	81.0	2.33	52.7
240	20	69.6	0.73	19.7	80.3	3.61	48.7
480	15	76.4	1.19	22.1	77.9	5.33	42.6
840	11	85.5	1.82	24.4	75.6	9.05	35.3
Ageing at 105°C							
24	23	67.5	0.66	17.3	82.7	2.24	52.6
48	18	71.8	0.87	20.0	80.0	2.96	50.1
120	14	78.5	1.35	22.3	77.7	5.57	42.7
240	9	91.0	2.11	29.9	70.1	12.40	32.6
480	6	123.0	4.40	36.2	63.8	19.70	24.6
Ageing at 125°C							
24	20	73.8	1.30	21.6	78.4	3.49	46.6
48	11	88.0	2.12	26.3	73.7	7.82	37.7
72	7	102.0	2.84	31.8	68.2	11.0	30.5
96	6	117.5	3.96	33.5	66.5	16.60	23.3

Note: Pen = Penetration, SP = Softening Point, PI = Penetration Index, G= Complex Modulus, and δ = Phase Angle*

Penetration vs. ageing time

Figure 1 shows a plot of penetration against the time taken for ageing the bituminous mixtures to the desired levels. The model used for fitting the curves is indicated on the chart. It can bee seen that ageing at 85ºC is only likely to produce sufficiently aged binder (target penetration of 3dmm) after very long ageing times, if at all and is not a suitable ageing protocol for producing the desires RAPs. At 105ºC the fitted curve indicates that such severe ageing could probably be attained after long ageing times (about 1000hours) but the upper 95% confidence interval indicates possibly very much longer ageing times would be required.

The results for ageing mixtures at 125ºC indicate a relatively short ageing time, by comparison (about 190 hours). Both the upper and lower 95% confidence intervals indicate reasonable laboratory times for the target ageing.

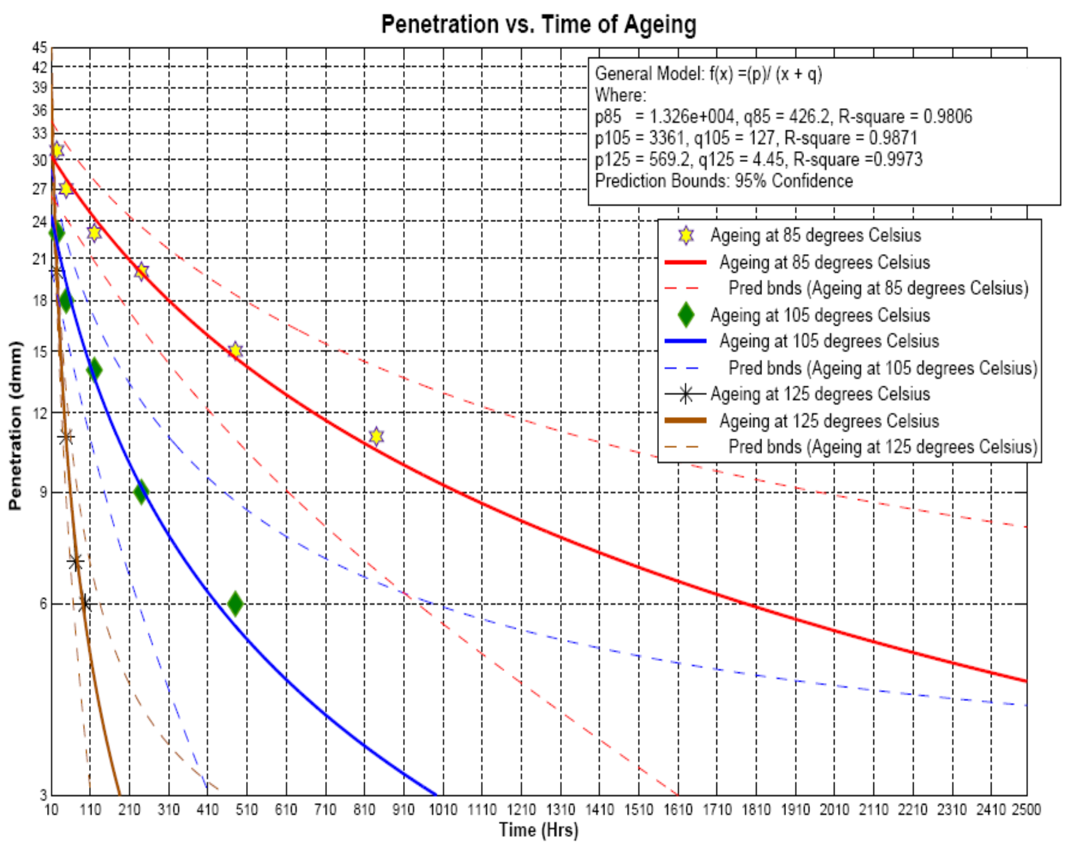

Fig. 1. Penetration of Recovered Aged Binders vs. Time of Ageing

Figures 2 and 3 show the temperature susceptibility of the binders recovered from the mixtures. Figure 2 reveals that the binders aged at 85ºC and 105ºC have similar temperature susceptibilities throughout the range. The binder aged at 125ºC showed a temperature susceptibility that diverged slightly from the other two ageing protocols at higher penetration values. As the binder hardens (into the range of interest for this study), the temperature susceptibility is similar to that for the other two protocols. In Figure 3 the binders from the three ageing protocols displayed similar temperature susceptibilities throughout the range.

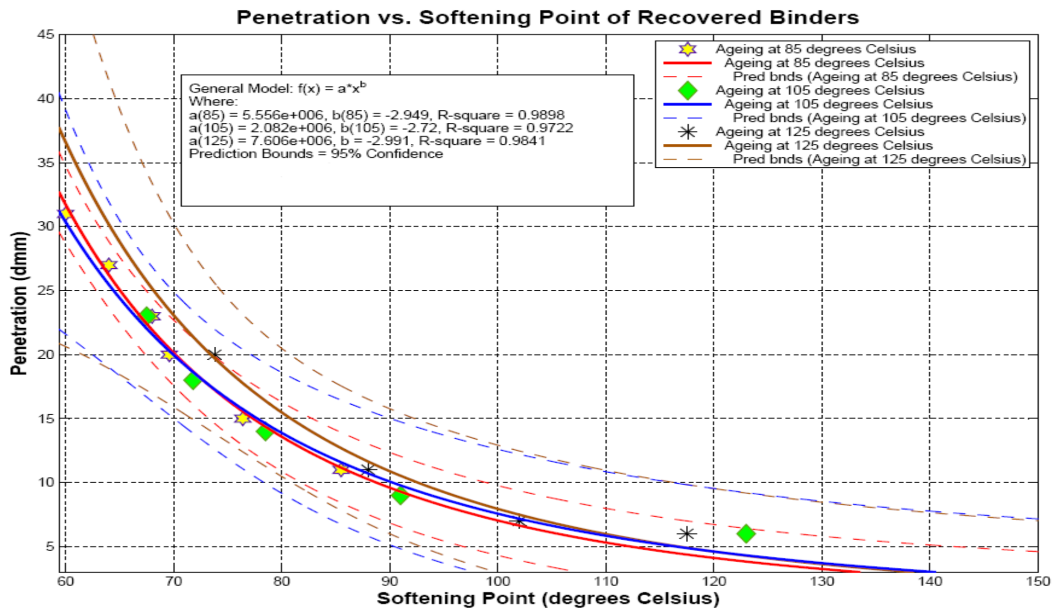

Fig. 2. Temperature Susceptibility of Recovered Binders (Penetration vs. Softening Point)

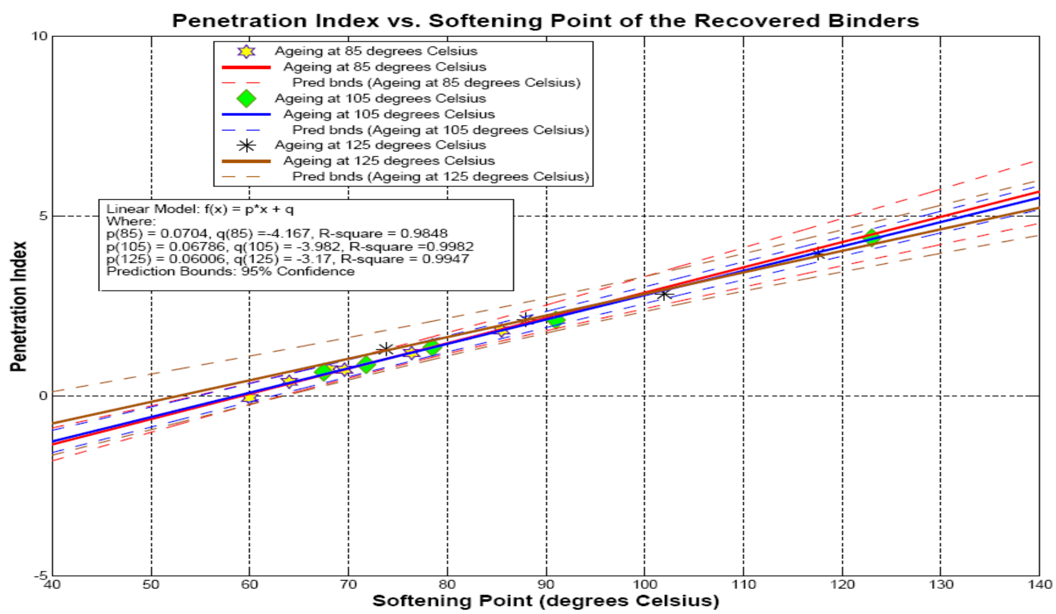

Fig. 3. Temperature Susceptibility of Recovered Binders (Softening Point vs. Penetration Index)

Chemical composition of the recovered bituminous binders

Results in Tables 1 and 2 generally confirm other researchers' findings that as bitumen ages, the asphaltene content increases at the expense of the maltenes, i.e. maltenes reduce in content [Read and Whiteoak, 2003]. In Figure 4, there were slight variations in the chemical compositions of the binders above a penetration value of 15dmm in which the aged binder at 125ºC displayed a noticeable difference of more than 10% in its value when compared to the binder aged at 85 ºC (BS 2000: Part 143:2004 recommends not greater than 10% variation in value). The aged binder at 105ºC showed this behaviour above the penetration value of 22.5dmm. The chemical compositions below these points were similar to that of the binder aged at 85ºC.

Figure 5 shows a dissimilar behaviour for the binders compared to Figure 4. At a temperature of about 100ºC and above (at which penetration is about 7.5dmm and lower), the binders displayed varying chemical compositions. Considered alongside Figure 2, the implication is that, when aged binders from the three different ageing protocols were much softer, they have different chemical compositions, but as they harden, the chemical compositions assume the same state.

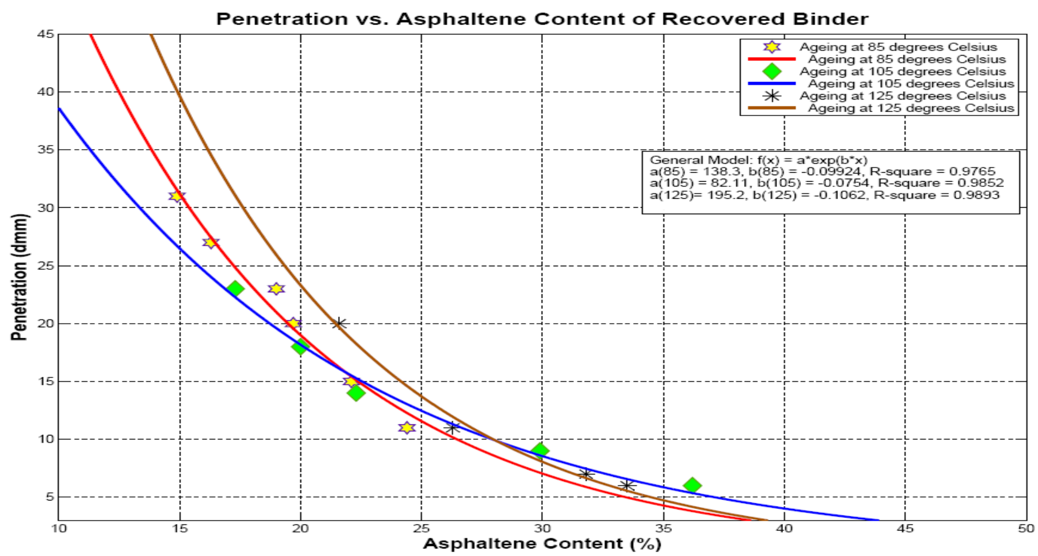

Fig. 4. Penetration of Recovered Binders vs. Asphaltene Content

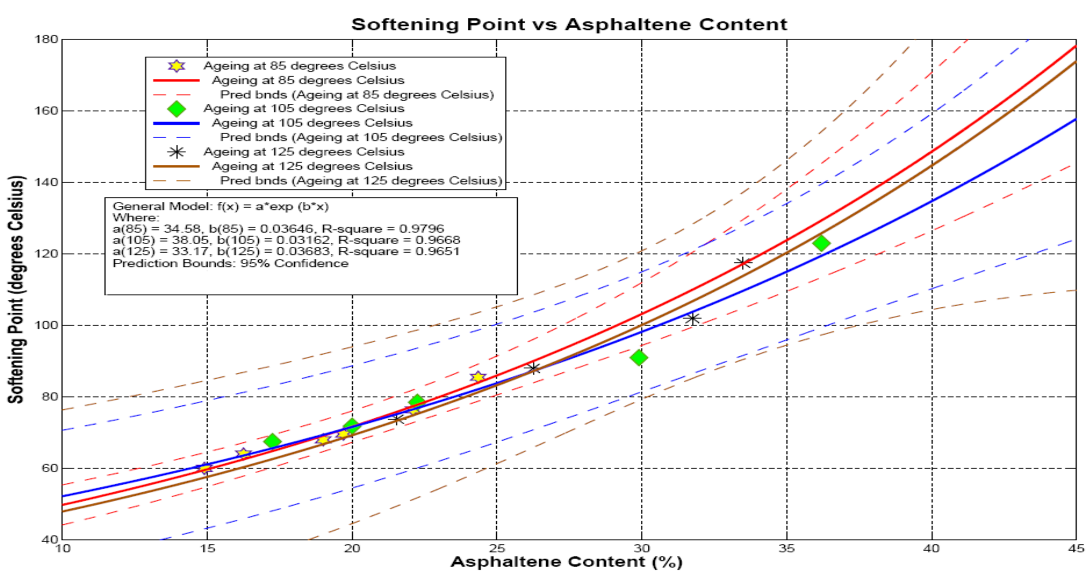

Fig. 5. Softening Point of Recovered Binders vs. Asphaltene Content

Rheological properties

The rheological properties of the RTFO and PAV aged bulk binders (over the durations indicated in Table 1) and that of the recovered binder from a 20mm DBM mixture (using granite aggregate and 40/60 paving grade Venezuelan binder obtained from a local quarry) have been considered along with the recovered binders from the laboratory mixtures aged at 85ᵒC, 105ᵒC and 125ᵒC (over the hours stated in Table 2). In order to allow for direct comparison, binders with similar penetration values though with different ageing protocols were examined. For example, Figure 6 shows the viscoelastic responses of recovered aged binders with penetration values of 23dmm, but from different ageing protocols of 85ºC (mixtures ageing over 120hrs) and 105ºC (mixtures ageing over 24hrs) and the PAV at 100ᵒC (bulk binder ageing over 25hrs). It is obvious from the figure that they all have similar viscoelastic responses, more importantly, they all remained thermorheologically simple just as the parent 53dmm penetration original binder.

Similarly, Figure 7 shows the viscoelastic responses of recovered aged binders with penetration values of 20dmm, but from different ageing protocols of 85 ºC (mixtures ageing over 240hrs) and 125ºC (mixtures ageing over 24hrs), PAV at 100ᵒC (bulk binder ageing over 15hrs) and recovered binder from 20mm DBM. The chart here reveals that there is a slight difference in the viscoelastic responses (slight shift in the trend of the curves) of the binders at this level, most especially between the PAV aged binder and the recovered binder from mixtures aged at 125ᵒC, at the lower frequencies and complex shear modulus. All the plots still indicate that the binders remained thermorheologically simple.

Figure 8 shows that bituminous mixtures aged at 85 ºC and 125 ºC have recovered binders of similar viscoelastic responses and they both remained thermorheologically simple. The penetration at this level is 11dmm. This observation is important to the theme of this investigation as it shows that there is little or no difference in the dynamics of ageing (or properties of the severely aged binder) in bituminous mixtures aged to a low penetration level, irrespective of whether it was at a temperature of 85ᵒC or 125ᵒC. Significantly, the findings also show that there is no departure from the thermorheological simplicity.

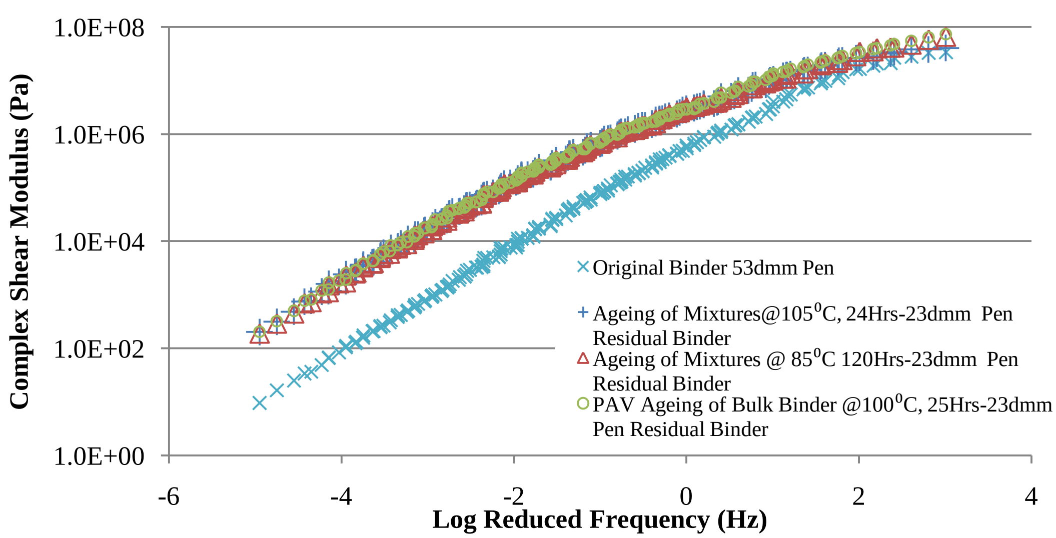

Fig. 6. Complex Modulus Master Curves of 23dmm Pen Binders Aged Differently Compared to the Parent 53dmm Pen Binder

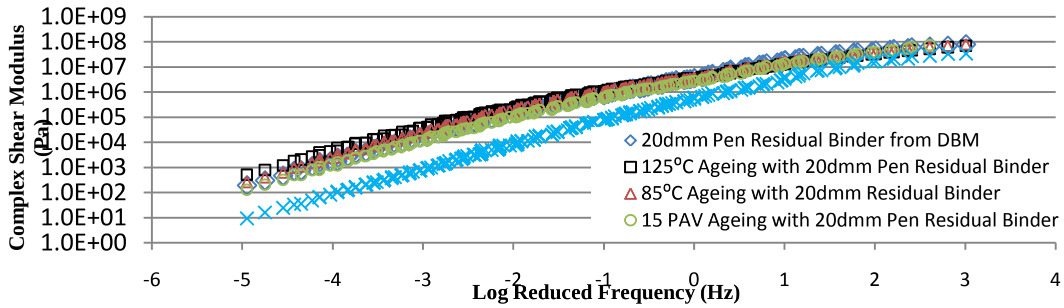

Fig. 7. Complex Modulus Master Curves of 20dmm Pen Binders Aged Differently Compared to the Parent 53dmm Pen Binder

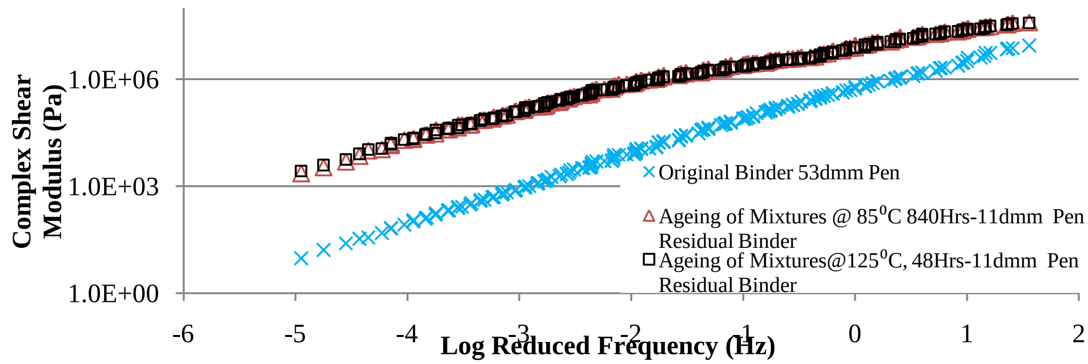

Fig. 8. Complex Modulus Master Curves of 11dmm Pen Binders Aged at 85°C and 125°C Differently Compared to the Parent 53dmm Pen Binder

Comparison of rheological and physicochemical properties of binders

Figures 9 to 11 compare rheological properties (complex modulus at 0.4Hz and 25°C) with physicochemical properties of the binders recovered from aged mixtures and the bulk binders aged under the RTFO and PAV protocols. It can be seen from these Figures that the binder, over the tested ranges and irrespective of the protocol by which it is aged, shows similar trends whether in mixture or as bulk binder.

CONCLUSIONS

Based on this study, the following conclusions and recommendations can be drawn from the study presented in this paper:

- Generally, as the binder used in this study (straight run paving grade binder 40/60 of Venezuelan origin with a penetration of 53dmm) ages, complex modulus, asphaltene content and softening point values increase while penetration values reduce irrespective of the ageing protocol i.e. either as bulk binder ageing or mixture ageing just in line with other researchers' findings.
- Ageing bituminous materials at 85°C over 120hrs (5days) in the forced draft oven did not yield the severity of ageing (below 6dmm) required to study heavily aged pavements typical of the tropics. This standard protocol yielded a residual binder of 23dmm penetration for the original 53dmm penetration binder used. However, it was able to age to 11dmm penetration after 840hrs (35days).

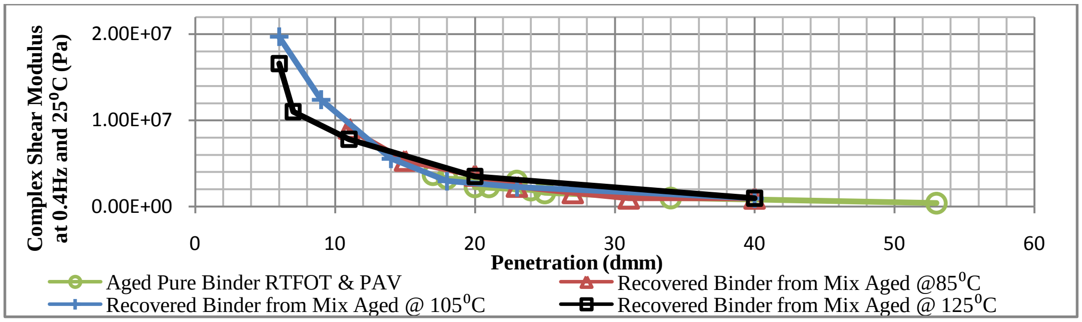

Fig. 9. A Comparison of the Complex Modulus and Penetration of PAV and RTFO Aged 53dmm Pen Binder with that of Recovered Binder

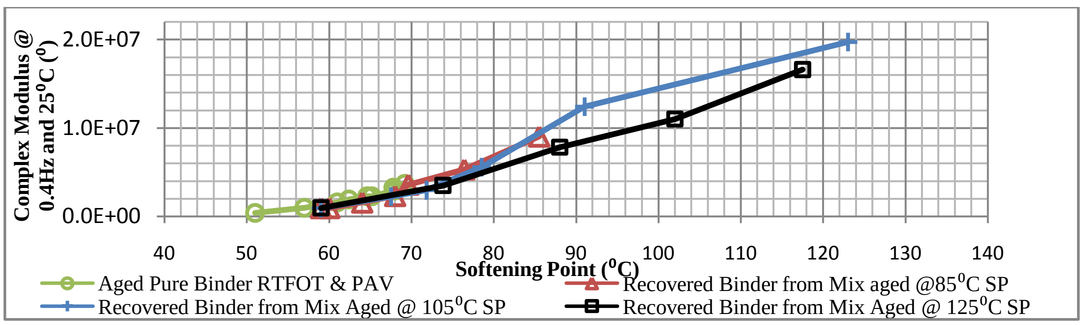

Fig. 10. A Comparison of the Complex Modulus and Softening Point of PAV and RTFOT Aged 53dmm Pen Binder with that of Recovered Binder

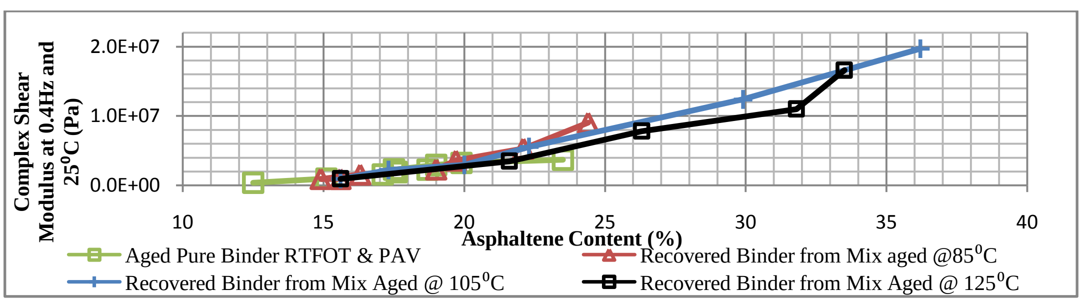

Fig. 11. A Comparison of the Complex Modulus and Asphaltene Content of PAV and RTFO Aged 53dmm Pen Binder with that of Recovered

- Ageing bituminous mixture at a higher temperature of 125°C accelerates ageing, such that it only took 48hrs to reach a penetration of 11dmm compared to 840hrs (35days) for the standard protocol and thus saves laboratory time.
- The rheological properties and temperature susceptibilities of less severely aged binders (20dmm penetration and above in this study), seem to differ slightly. This was most obvious between the PAV aged binders and the recovered binders of mixtures aged at 125°C.
- The properties of more severely aged binders are independent of the ageing protocol.
- It is recommended that PAV ageing of binders for durations longer than 35hrs should be conducted and properties be compared to the most severely aged binders in this study.

- The measurement of chemical composition should be extended beyond asphaltene content to a full SARA analysis, in order to further explore the relationship between softening points, penetration values and rheological properties of severely aged binders.

ACKNOWLEDGEMENTS

Mr. Oke is grateful to the University of Ado-Ekiti, Nigeria and University of Nottingham, UK for a PhD scholarship. This work received support from Nynas Bitumen, UK, Cliffe Hill Quarry, UK and Longcliffe Quarries, UK. The particular help of Dr Dennis Day of Nynas Bitumen is gratefully acknowledged. Sincere thanks also go to Mr. Richard Blakemore and M.r Lawrence Pont and their respective teams, all of NTEC, University of Nottingham, for their assistance in testing some of the materials mentioned in this paper.

REFERENCES

ARRA (2001). "Basic Asphalt Recycling Manual." *Asphalt Recycling and Reclaiming Association (ARRA)*, USA.

Asphalt Academy, (2009). "Technical Guideline: Bitumen Stabilised Materials, A Guideline for the Design and Construction of Bitumen Emulsion and Foamed Bitumen Stabilised Materials", TG 2, Second Edition, *Asphalt Academy*, Pretoria.

Airey, G.D. (2003). "State of the Art Report on Ageing Test Methods for Bituminous Pavement Materials." *The International Journal of Pavement Engineering*, Vol. 4 (3) September 2003, pp165-176.

Bahia, H. U., and Anderson, D. A. (1995). "The SHRP binder rheological parameters: Why are they required and how do they compare to conventional properties?" *Transportation Research Record 1488*, Transportation Research Board, Washington, D.C., 32–39.

Bell, C.A. (1989). "Summary Report on Aging of Asphalt-Aggregate Systems". SHR-A/IR-89-004, *Strategic Highway Report Research Program*, National Research Council.

de la Roche, C., Van de Ven, M., Van den Bergh, W., Gabet, T., Dubois, V. and Grenfell, J. (2009). "Development of a laboratory bituminous mixtures ageing protocol." *Advanced Testing and Characterization of Bituminous Materials*, Lois, d. Q., Partl, M., Scarpas, T. and Al-Qadi, I. (eds), Taylor and Francis Group, London.

Nnenna, O.J. (2003). "Highway Maintenance in Nigeria: Lessons from Other Countries." *Central Bank (of Nigeria) Research Department Occasional Paper Series*, CBN, Abuja.

PIARC (2008). "Review of the Growth and Development of Recycling in Pavement Construction." *World Road Association*, Cedex.

Read, J. M. and Whiteoak, D. (2003). "*The Shell Bitumen Handbook.*" Fifth Edition, Shell Bitumen, UK, Chertsey.

Scholz, T.V. (1995). "*Durability of Bituminous Paving Mixtures.*" PhD Thesis, School of Civil Engineering, University of Nottingham, Nottingham.

Smith, H.R. and Edward, A.C. (2001). "Recycling of Bituminous Materials, DFID Project Report PR/INT/225/01", Project No. R6474, *TRL Ltd*, UK.

UNECA (2007). "The Transport Situation in Africa." *Fifth Session of the Committee on Trade, Regional Cooperation and Integration*, Addis Ababa.

Wu, J., Lepercq, E. and Airey, G. D. (2007). "Ageing of bitumen in bulk versus the ageing in asphalt mixture", *Advanced Testing and Characterization of Soil Engineering Material*, Lois, d. Q., Partl, M., Scarpas, T. and Al-Qadi, I. (eds), Taylor and Francis Group, London.